

Chemical Synthesis: Martin Method

Used to synthesize gold nanoparticles ranging from 1-10nm

Protocol:

Perform all steps in FUME HOOD.

1. Measure 20 mL of ddH₂O into 50 mL graduated cylinder and pour into 125mL Erlenmeyer flask.
2. Keep some extra ddH₂O on ice for later steps in a falcon tube.
3. Weigh out 1.96 mg (0.00196 g) of chloroauric acid trihydrate (HAuCl₄*3 H₂O) solid on a weigh-boat.
 - CAUTION: oxidizes quickly - do not expose to air for longer than required
 - Do NOT use metal scoopula - use plastic or wood to transfer the solid
 - LIGHT SENSITIVE - use for short duration under light conditions
 - Note: chloroauric acid is expensive - try not to be wasteful!
4. Using a micropipet, quickly add ~40 uL of the ddH₂O from the flask to the crystals of chloroauric acid trihydrate on the weigh-boat. This should dissolve the solid to generate a yellow mixture on the weigh-boat. Then use the micropipette to transfer the yellow liquid to a 1.5 mL microfuge tube.
 - By avoiding transfer of the solid form of HAuCl₄, the possibility of loss of sample is lessened. Transfer as a liquid is preferable.
 - Seal the tube rapidly to limit air exposure of the chemical.
5. Micropipet the HAuCl₄ solution directly into the flask and seal the opening with aluminum foil.
6. Add stir bar to flask and mix on plate to allow the solid HAuCl₄ to dissolve.
 - Nanoparticle synthesis occurs at this step.
 - Having the flask opening covered with aluminum foil prevents the escape of fumes from the synthesis reaction.
7. Weigh out 1.47 mg (0.00147 g) of trisodium citrate dihydrate solid.
8. Add the trisodium citrate dihydrate to the mixture in the flask with stirring.
9. On ice, prepare 0.1 M sodium borohydride (NaBH₄) solution.
 - Pipet 0.65 mL (650 uL) of cold ddH₂O into a 1.5mL microfuge tube kept on ice.
 - Weigh out 2.46 mg (0.00246 g) of NaBH₄.
 - Put the measured NaBH₄ into the microfuge tube containing ddH₂O and resuspend by pipetting until the solid is fully dissolved (keep on ice throughout).
10. Add 0.6 mL (600 uL) of the cold 0.1 M NaBH₄ solution into the mixture in the Erlenmeyer flask while stirring vigorously on the stir plate. - NOTE: the solution should turn dark red immediately - indicating particle formation.
11. The solution is then tested for presence of nanoparticles using UV spectrophotometry
 - Expected absorbance peak between 510-525 nm.
12. Once nanoparticles are confirmed in solution:
 - Weigh out 0.15 g of L-cysteine solid.
 - Add the L-cysteine to the nanoparticle solution with stirring for ~10 min.
13. Collect nanoparticles by pelleting using centrifugation at 1,000 RPM at room temperature for 50 minutes.
14. Store the solution in 4°C fridge.

Source: Nan, J., Xiao-Yu, Y., Guo-Liang, Y., Ling, S., Jing, L., Wei, G., Ling-Jun, D., *et al.* 2015. "Self-repairing"nanoshell for cell protection. *Chem Sci*, 6: 486-491.